



## Original Article

# Synthesis and Characterization with Antimicrobial Studies of mixed Curcumin Schiff Base-L-dopa with Divalent Cations Complexes

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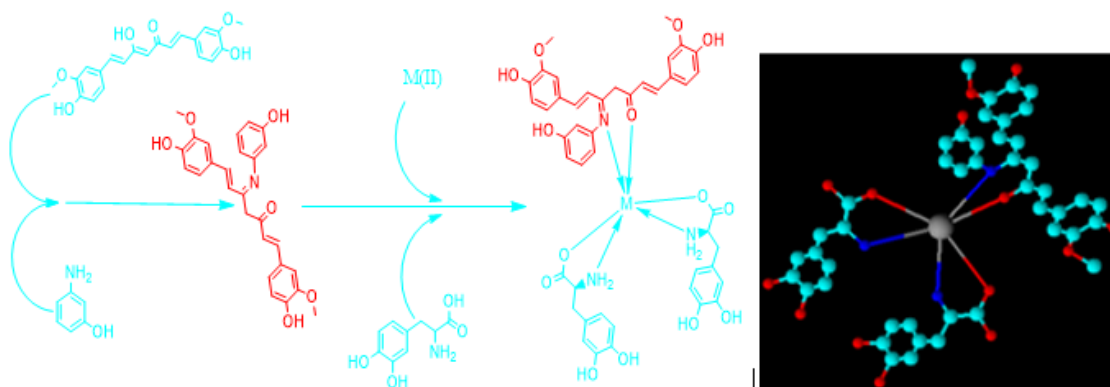
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## ABSTRACT

In the present article, mixed ligand metal (II) complexes have been synthesized with Schiff base (1E, 5Z, 6E)-1,7 bis (4-hydroxy-3-methoxyphenyl)-5-(3-hydroxyphenyl) imino) hepta-1,6-dien-3-one derived from Curcumin and 3-aminophenol as primary ligand and L-dopa as a secondary ligand. The Schiff base act as bidentate and arrange to the metals through the azomethine (C=N) nitrogen and (C=O) oxygen atom. The mode of bonding of the Schiff base has been affirmed on the infrared by the UV-Visible,  $^1\text{H}$ , and  $^{13}\text{C}$  NMR spectroscopic techniques. The magnetic susceptibility and the UV-Vis data of the complexes propose octahedral geometry around the central metal ion. The information appears that the complexes have the structure of [L-M-(L-dopa)] system, where M=Mn(II), Ni(II), Cu(II), Zn(II) and Cd(II). The low conductance of all complexes supports the non-electrolytic nature of the complexes. The biological activity of the complexes was screened against bacteria, (*Klebsiella* and *P. Staphylococuse* and gives a good results anti-fungus *C. Albicans*). All complexes possess the biological activity which has the same activities, and good antimicrobial activity that displays higher activities against fungus compared with bacteria.

## GRAPHICAL ABSTRACT



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## Introduction

Curcumin compounds have a great combination with drug or amino acids glycine-Curcumin form compounds, inhibits the growth of fungal and bacterial strains after complexation [1, 2]. The complexation behavior of di-ketones in metal complexes give information of the structural, nature of coordination, and spectral data [3, 4]. Schiff base complexes played a central role in bioinorganic chemistry because of their biological importance and industrial [5, 6]. O- and N-donors tridentate Schiff bases complexes have been reported to exhibit different activities on DPPH radicals [7]. Abu-Dief *et al.* synthesized and characterized Schiff bases-transition metal-complexes by the condensation of 5-bromosalicylaldehyde and six L- $\alpha$ -amino acids [8-10]. The theoretical study of curcumin-M = Nickel(II), Copper(II), and Magnesium(II) complexes performed by density-functional theory [11].

The most objective of this inquiry was to synthesize and characterize the Schiff base, and its mixed ligand metal(II) complexes with amino acid L-dopa and, decide their antibacterial activity in the present work.

## Materials and Methods

### Materials and physicochemical analyses

All the chemicals were obtained from Sigma-or BDH and utilized without assistance purification. Curcumin, L-dopa, DMSO, and C<sub>2</sub>H<sub>5</sub>OH were Analar grade (BDH).

The FT-IR spectra were recorded in 400–400 cm<sup>-1</sup> region with 8300-Shimadzu spectrophotometer, Magnetic susceptibility. Metal determination was done with spectrophotometer (A.A-160). The mass spectrum was recorded by MS Model: 5973 spectrometer, Elemental microanalysis C.H.N-2400 elemental analyzer. The NMR spectra of proton and carbon were recorded for (L) in a solvent DMSO, by using a device (INOVA-500-MHz) in  $\delta$  (ppm). The UV spectra (10<sup>-3</sup> M) by (Shimadzu U.V 160-A),  $\lambda_m$  10<sup>-3</sup> mol/L in DMSO were reached by Ino.Lab.720 digital conductivity.

### Synthesis of Schiff base

The synthesis of Schiff base (L) was made concurring to the methods described in the literature.<sup>12</sup> In 100 mL flask, the (L) was prepared by curcumin condensation, of which (3 mmol, 1.104 g) was dissolved in (25 mL) boiling ethanol. Then, NaOH (3 mmol, 0.119 g) was added and dissolved in (10 mL) of ethanol. After that, 3-aminophenol (3 mmol, 0.327 g) was dissolved in (30 mL) ethanol and added to the mixture with the constant stirring. The reaction mixture was refluxed for 12 hours at 70 °C until the completion of the reaction and the response and also, the advance of the response was checked by TLC. After the reaction was finished, the drops of glacial acetic acid were added (Scheme 1). The yellow-colored compound was precipitated. The yellow-colored accelerated compound was washed with hot ethanol, recrystallized to urge an unadulterated test, and dried desiccator in CaCl<sub>2</sub>.

### Synthesis of mixed complexes of Schiff base and L-dopa

The mixed complexes (**1-5**) were synthesized by reacting the (L) and L-dopa with the Mn(II) (**1**), Ni(II) (**2**), Cu(II) (**3**), Zn(II) (**4**) and Cd(II) (**5**) ions according to the literature method [12], as depicted in Scheme 1.

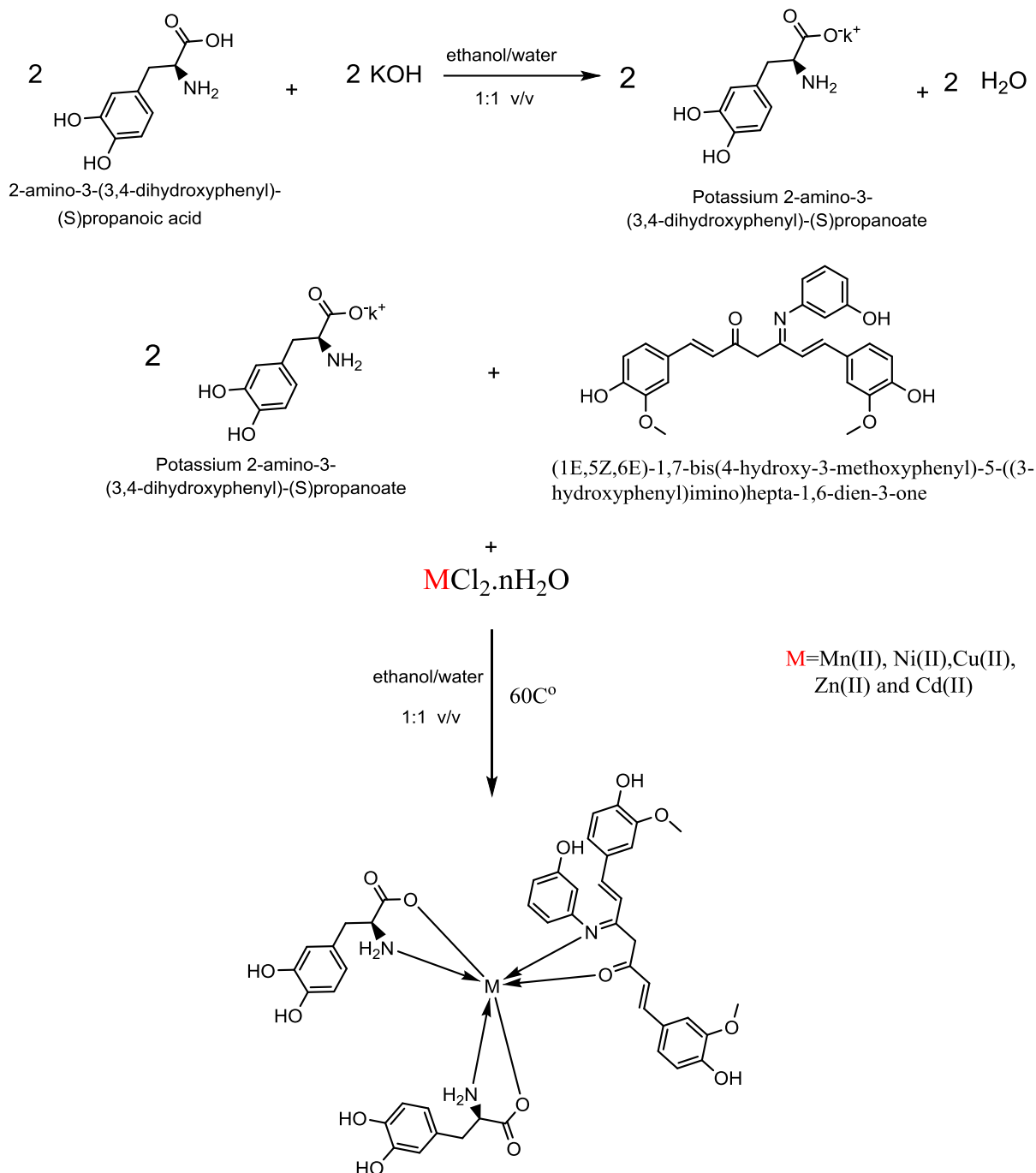
## Results and Discussion

### Characterization of the Schiff base (L)

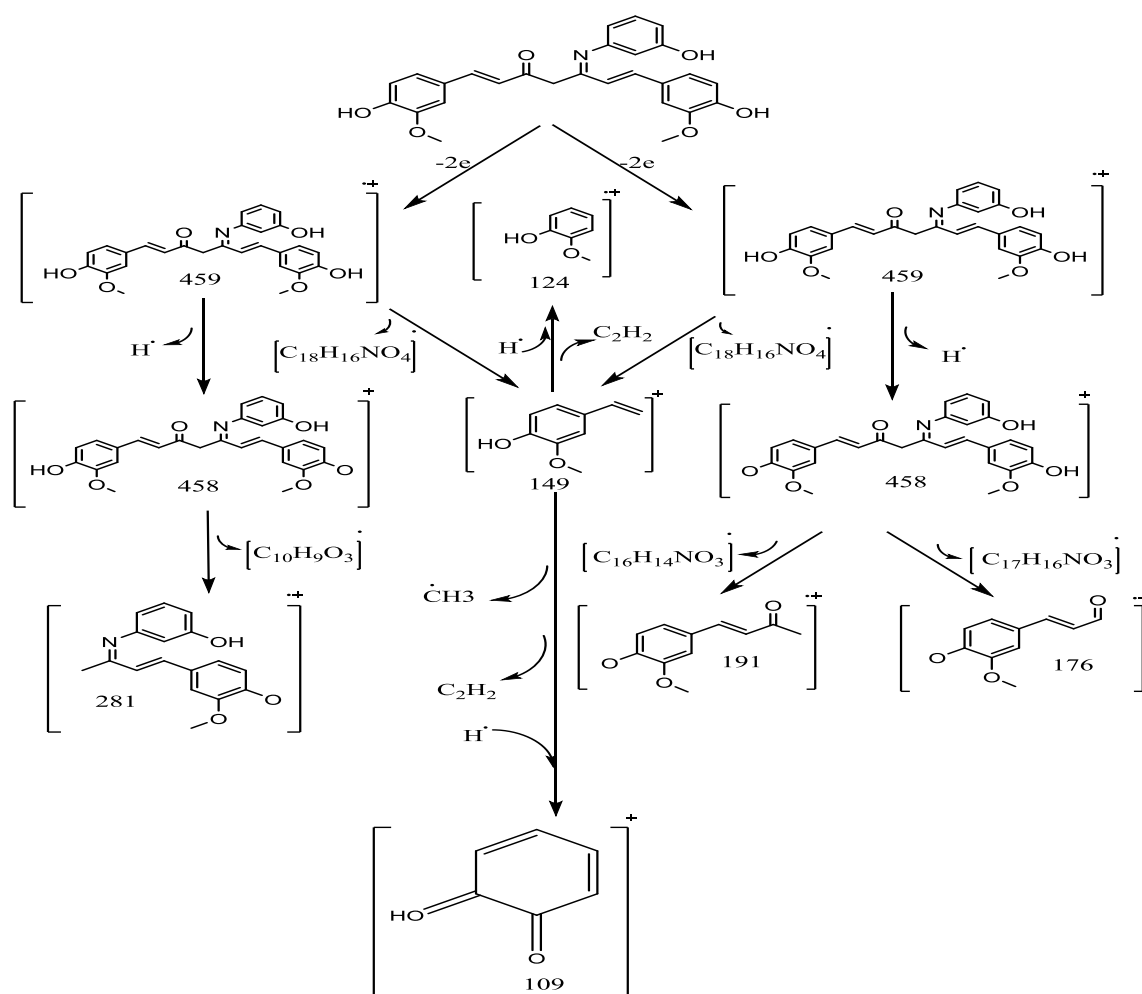
The yield of the prepared (L) was 87% (solid, color: orange, m.p. 295 °C). FT-IR (KBr, cm<sup>-1</sup>), as displayed in Figure S1 (Supporting information), 3242  $\nu$  (O-H), 1610  $\nu$  (C=N), 1701  $\nu$  (C=O), 1589  $\nu$  (C=C), 1029  $\nu$  (C-O-C), and 1126  $\nu$  (OCH<sub>3</sub>) [13-16]. UV-Vis, 10<sup>-3</sup> mol L<sup>-1</sup> in DMSO,  $\lambda_{max}$  nm: 284 and 364 were assigned to ( $\pi \rightarrow \pi^*$ ) and ( $n \rightarrow \pi^*$ ) transitions, respectively. <sup>1</sup>H-NMR (DMSO, 400 MHz), in Figure S2 (Supporting information), 8.8- 8.65 ppm  $\delta$  (s, 1H, OH) phenol, displays 8.8-8.65 ppm  $\delta$  (OH) phenol, a multiple in the region  $\delta$  (m, 7H, ArH). 6.41-7.35 ppm was corresponded to the aromatic protons other than the over peaks, the chemical move values of  $\delta$  (-CH=CH-) aliphatic 4.5, 5.3, 3.45 (H-

CH<sub>2</sub>), and 3.7 (H-OCH<sub>3</sub>), as illustrated in Figures S3 and 4 ([Supporting information](#)) [17, 18]. The <sup>13</sup>C-NMR spectrum of (L) is indicated in Figure S3 ([Supporting information](#)). The <sup>13</sup>C-NMR peaks for the azomethine carbon atom and phenolic group carbons of the coordinated Schiff base ligand were observed at  $\delta$  =166.5 ppm and 158.84 ppm, respectively [13, 17].

The mass spectrum of (L), in Figure S4 ([Supporting information](#)), showed the parent ion peak at M/Z [C<sub>27</sub>H<sub>25</sub>NO<sub>6</sub>] = 459 (M + which is steady with the atomic weight of the Schiff base. The other fragments, their relative abundances, and fragmentation pathways data are depicted in [Scheme 2](#) and strongly confirmed the formation and the structure of the Schiff base.



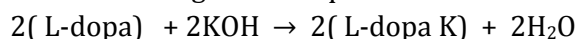
**Scheme 1:** General scheme for the synthesized complexes



**Scheme 2:** Proposed mass spectral fragmentation pattern of the (L)

#### Characterization of [L- Metal-L-dopa] Complexes

All the complexes were destined reacting the metal chlorides  $MCl_2 \cdot nH_2O$  and the ligands by utilizing 1:2:1 mole ratio, i.e. metal: two moles of L-dopa K: Schiff base. The synthesis of  $[M(L)(L-dopa)_2]$  complexes may be represented as shown in the following overall equation:



$MCl_2 \cdot nH_2O + L + 2(L-dopa K) \rightarrow [M(L)(L-dopa)_2] + 2KCl + nH_2O$  Where, L=Schiff base ( $C_{27}H_{25}NO_6$ ) accrued from condensation of (Curc) and 3-aminophenol as a primary ligand, L-dopa as a secondary ligand.

L= deprotonated of (L-dopa)

As listed in Table 1, the melting points (m.p) of the complexes were higher than that of the ligands, demonstrating that the complexes are steadier than the ligands thermally. The solubility of the complexes was examined in different solvents appeared that all complexes are dissolvable in all solvents. The test for chloride

particle with  $AgNO_3$  arrangement was negative (Nil%) demonstrating that there is no (Cl-) outside to the coordination circle of the central metal. The calculated and exploratory values of metal percentage (M%) in each complex are in sensible agreement [17]. The formula weights, elemental analysis of CH.N data, and melting points are presented in Table 1. The molar conductance  $\Lambda_m$  values for the complexes were found between 2.10 and 19.00 ( $\Omega^{-1} cm^2 \cdot mol^{-1}$ ) in DMSO indicating their non-electrolytic nature [18].

#### FT-IR spectra

The important FT-IR data of all complexes are presented in Table 2. The FT-IR spectra of the complexes, are similar and there are no major differences in their vibrational frequencies. Generally, as compared FT-IR spectra of the complexes with those of free L-dopa and (L), we observe shift of the most of bands to the lower or

higher frequencies. It is probably caused by coordination of two ligands [11-13].

**Table 1:** Elemental analysis, metal%, and melting pointsof the [(L-Metal-L-dopa)]

Complexes	Color	M.W	Yield (%)	m.p °C	Found (Calc.)%			
					C	H	N	Metal % By AAS
[Mn(L)(L-dopa)2]	Dark brown	906.78	76	>250	(59.60) 60.10	(4.98) 5.10	(4.63) 4.33	(6.06) 7.22
[Ni(L)(L-dopa)2]	Light brown	910.54	66	>250	(59.36) 60.00	(4.98) 4.50	(4.61) 4.33	(6.45) 7.00
[Cu(L)(L-dopa)2]	Greenish brown	915.39	87	>250	(59.04) 60.00	(4.95) 4.88	(4.59) 4.44	(6.94) 7.20
[Zn(L)(L-dopa)2]	Dark brown	917.25	80	>250	(58.92) 60.00	(4.94) 4.77	(4.58) 4.40	(7.13) 6.88
[Cd(L)(L-dopa)2]	Light brown	964.26	77	>250	(56.05) 55.55	(4.70) 4.30	(4.36) 4.40	(11.6) 10.16

The blue and red shifts of observed bands are due to the formation of M-  $\nu$  (C=N-) and  $\nu$  (C=O) coordination bonds. The shifts in complexes suggest that the bond formation of the metal. The oxygen of the ketone part binds metal cations [11]. De-protonation of L-dopa is neighborhood in nature, for all locales considered the changes in geometry and charge thickness are localized within the locale where the deprotonation took place. In any case, the impact of carboxyl gather deprotonation is transmitted to the C-NH<sub>2</sub> locale and leads to the collection of the abundance charge thickness on N iota in NH<sub>2</sub> group. This point is of extraordinary significance when concerned. The spectra of the complexes displayed two different absorption bands in the 1627 and 1427 cm<sup>-1</sup>, which correspond to the difference between the  $\nu$  asym (COO) and  $\nu$  sym

(COO) vibrational modes of the carboxyl groups, distinction  $\Delta\nu = [\nu \text{ asym}(\text{COO-}) - \nu \text{ sym}(\text{COO-})] > 200 \text{ cm}^{-1}$  falls in the range of 274–262 cm<sup>-1</sup>. This results for carboxyl group associated with monodentate manner coordination [14-16] with the metal ions. L-dopa acts as bidentate ligand coordination by the carboxyl oxygen COO  $\rightarrow$  and the  $\leftarrow$  NH<sub>2</sub> nitrogen atom of amino group with the active donor atoms. Inclusion t of nitrogen in the coordination was backed by the appearance of the groups comparing  $\nu$  (C=N) and  $\nu$  (M-N) in 1512–1597 and 516–582 cm<sup>-1</sup>, which confirms coordination through the  $\nu$  (C=N-) nitrogen to the M(II) moiety. The bands at 516–595 cm<sup>-1</sup> were relegated to  $\nu$  (M $\leftarrow$ N). The new low intensity bands in the region of (432–482 cm<sup>-1</sup>) are attributed to  $\nu$  (M-O).

**Table 2:** Infrared spectral data for the mixed [L-M-L-dopa]

Ions Comp.	O-H phenolic	N-H <sub>2</sub>	C-H	C-H	COO	COO	C=N	C=C	M $\leftarrow$ N M-N	M-O
Mn (1)	3236	3471 3414	2943	2839	1616	1373	1512	1543	516	482
Ni (2)	3236	3464 3417	2970	2843	1612	1377	1512	1543	520	474
Cu (3)	3238	3471 3414	2970	2839	1616	1377	1597	1518	532	432
Zn (4)	3238	3479 3414	2974	2839	1616	1346	1565	1543	582	474
Cd (5)	3246	3475 3414	2977	2839	1598	1373	1512	1597	563	435

*Electronic spectra and magnetic moment*

All the UV-Vis spectra of the mixed L-dopa-Schiff base metal(II) complexes indicated the similar absorption spectra as the Schiff base which is either moved to the red or blue locale, as listed in Table 3. The (UV-Vis) spectrum for the (L-dopa) in DMSO ( $10^{-3}$  M) was characterized by 2-absorption bands at ( $\lambda_{\text{max}}$  277 nm,  $\pi \rightarrow \pi^*$ ) and ( $\lambda_{\text{max}}$  451 nm,  $n \rightarrow \pi^*$ ), within the transition aromatic ring and C=O, respectively, agreed with the reported data [18, 19]. The observed magnetic value of Ni(II) complex was 3.19  $\mu\text{B}$ , Cu(II) complex was 1.69  $\mu\text{B}$ , Mn(II) complex was 5.94  $\mu\text{B}$  which revealed an octahedral geometry [19], around the central metal ions. Zn(II) and Cd(II)  $d^{10}$  electronic configuration complexes are diamagnetic nature [20]. The electronic spectrum of the [Mn (L)(L-dopa)<sub>2</sub>] complex gave three bands at 285 nm and 364 nm were due to the intra-ligand transition, charge transfer, and the one at band at 864 nm was were due to  $6A_{1g} \rightarrow 4E_g$  transition [20, 21]. The electronic spectrum of [Ni (L)(L-dopa)<sub>2</sub>] complex given three bands at 284 nm and 366 nm are due to the intra-ligand transition, and charge transfer, and the one at 910 nm was as a result of [d-d] transition for  $3A_{2g} \rightarrow 3T_{2g}$  [21].

[Cu(L)(L-dopa)<sub>2</sub>] complex spectrum gave four bands. The bands at 287 nm and 362 nm are due

to the intra-ligand transition and C.T. and two at 826 nm and 910 were as a result of [d-d] transitions for  $3T_{1g} \rightarrow 3T_{2g}$  [20].

[Zn(L)(L-dopa)<sub>2</sub>] complex gave bands at 266 nm ( $37593\text{ cm}^{-1}$ ) and 429 ( $23310\text{ cm}^{-1}$ ) nm were due to (intra-ligand transition) and metal to ligand charge transfer (MLCT) transition, respectively [20].

[Cd(L)(L-dopa)<sub>2</sub>] complex gave bands at 285 nm ( $35087\text{ cm}^{-1}$ ), 345 nm ( $28985\text{ cm}^{-1}$ ), and 363 nm ( $27548\text{ cm}^{-1}$ ) were gave (intra-ligand transition) and (MLCT) transition, respectively [18].

An octahedral (goodness) geometry are proposed for all complexes based on its unearthy information and explanatory conductance, which displayed coordination number of six and may be defined as [M(L)(L-dopa)<sub>2</sub>], as demonstrated in Figure 1 [9, 11].

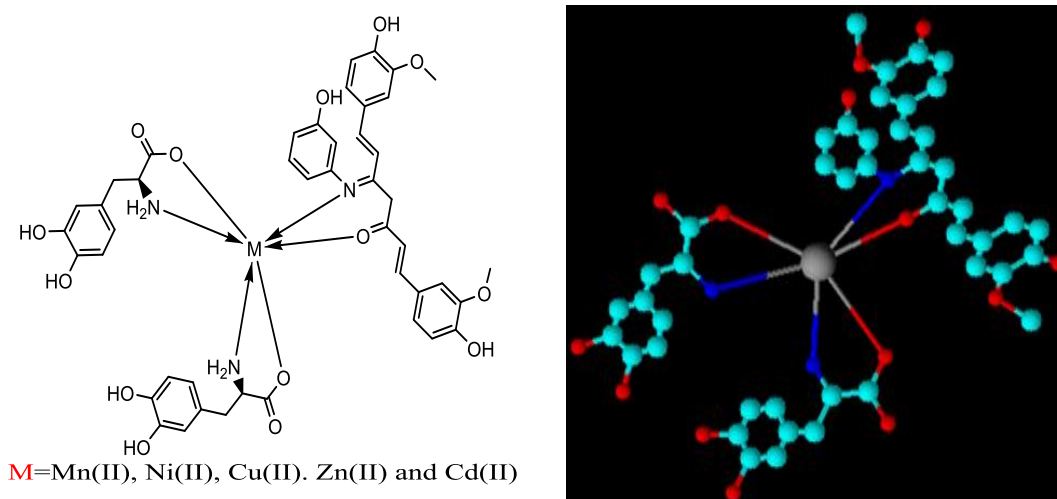
*The Proposed molecular structure for studying complexes*

Studying complexes on bases of the above analysis, the spectral observations suggesting the octahedral geometry for all the prepared complexes which exhibited coordination number six and may be formulated as [M(L)(L-dopa)<sub>2</sub>]. The general structure of the complexes is 3D, as is depicted in Figure 1.

**Table 3:** Data for electronic spectral of the [M (L) (L-dopa)<sub>2</sub>]

Complexes	$\lambda$ Max	$\nu$	$\epsilon\text{Mol}^{-1}.\text{L}.\text{Cm}^{-1}$	Assignments
[Mn(L)(L-dopa) <sub>2</sub> ]	285	35087	1161	Intra-ligand
	364	27472	895	Charge transfer
	864	11574	23	$6A_{1g} \rightarrow 4E_g$
[Ni(L)(L-dopa) <sub>2</sub> ]	284	35211	1356	Intra-ligand
	366	27322	944	Charge transfer
	910	10989	33	$3A_{2g} \rightarrow 3T_{2g}$
[Cu(L)(L-dopa) <sub>2</sub> ]	287	34843	1113	Intra-ligand
	362	27624	811	Charge transfer
	826	12106	4	$3T_{1g} \rightarrow 3T_{2g}$
[Zn(L)(L-dopa) <sub>2</sub> ]	266	37593	499	Charge transfer
	429	23311	1795	Charge transfer
[Cd(L)(L-dopa) <sub>2</sub> ]	285	35087	1259	Charge transfer
	345	28985	1742	Charge transfer
	363	27548	971	Charge transfer



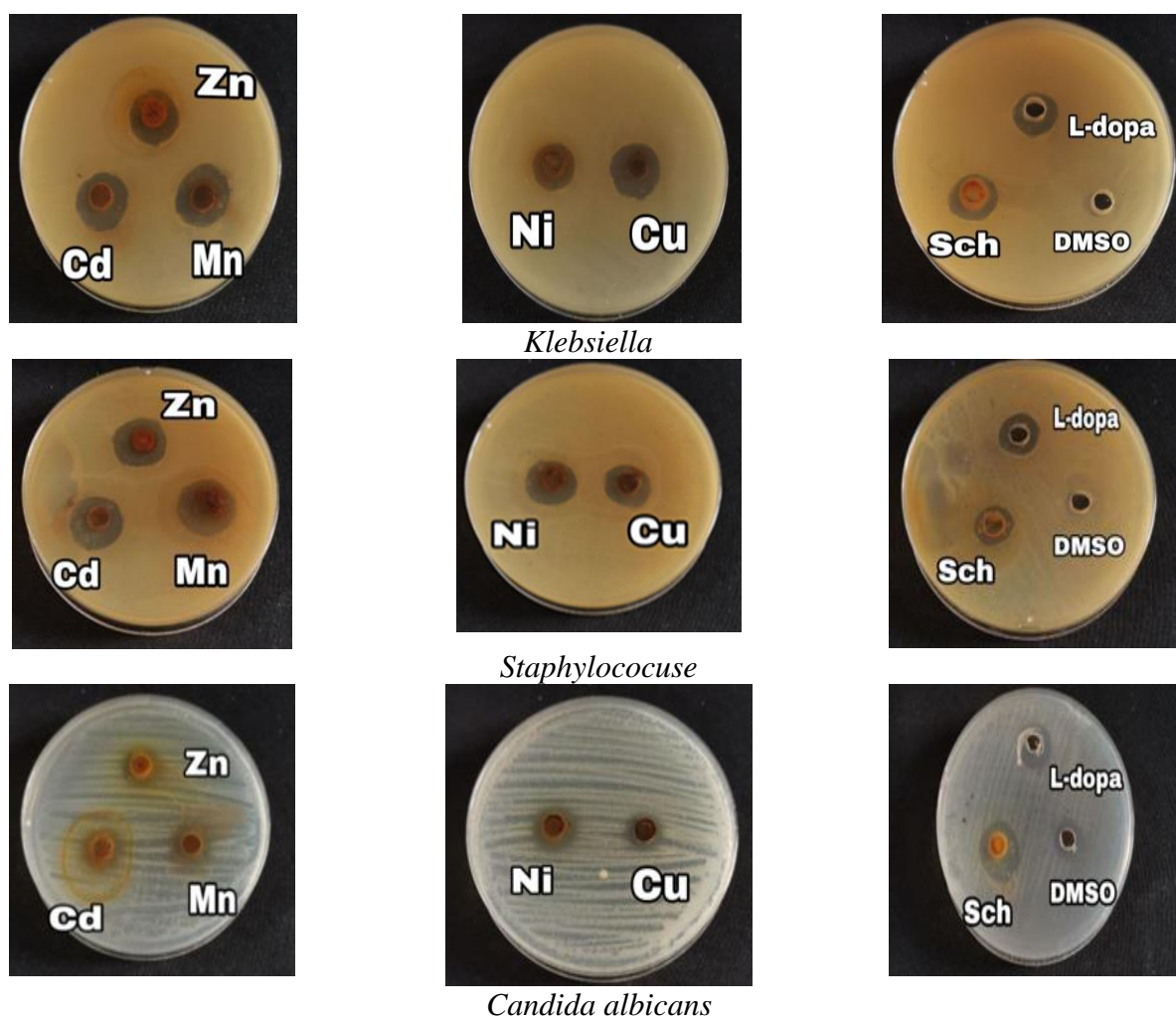


**Figure 1:** 3D molecular modeling proposed

### Biological activities

The tests were carried out on one Gram-positive (+) and one Gram-negative (-) bacterial strain concurring to the standard method [22]. All complexes are similar and there are no major

differences in their biological activity. The results were represented by using bar graphs in Figures 2 and 3. The inhibition zones were measured in millimeters (mm); high activity 11-15, very high activity  $\geq 16$ , and low activity for DMSO.

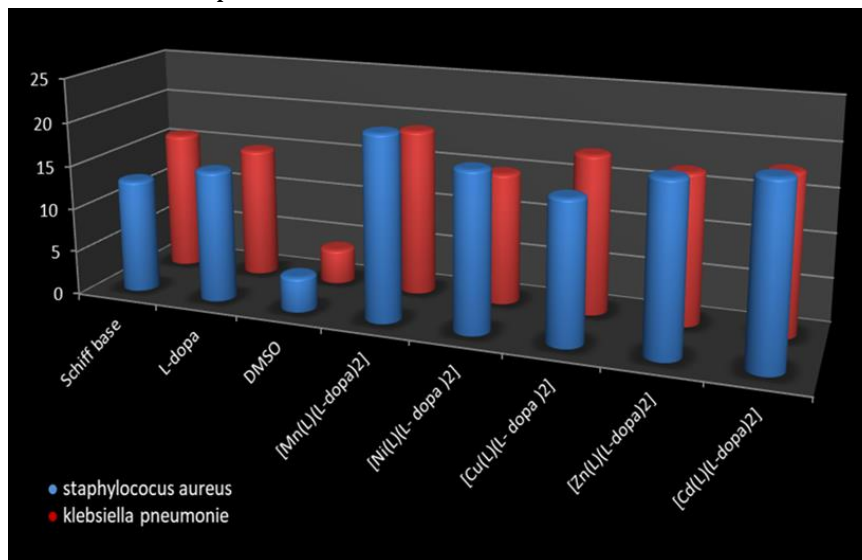


**Figure 2:** Plates zone of inhibition of compounds against *Staphylococuse*, *Klebsiella*, and *candida albican*

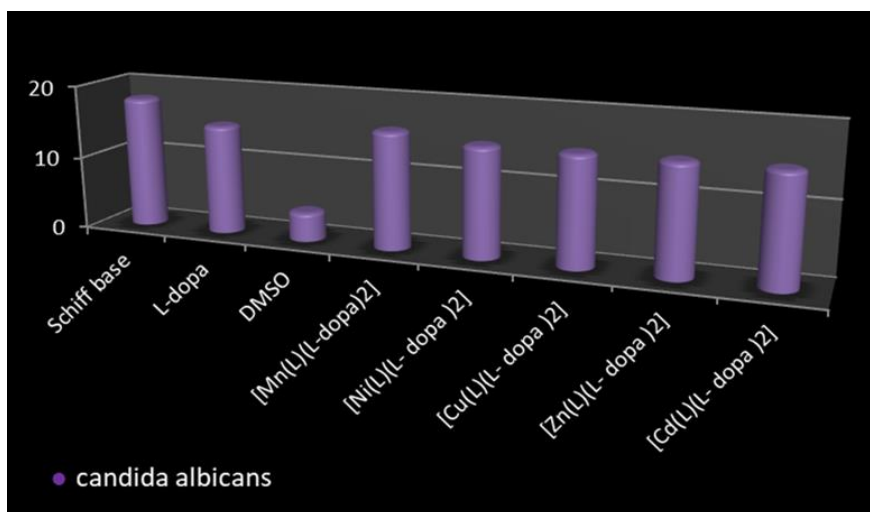
All the compounds showed a significant antibacterial activity against the tested bacteria, the ligands were found to be active against the bacterial strains and some complexes were more significant than the two ligands [11, 23].

The compounds were assessed on one strain of finding that all the test complexes initiated

antifungal movement against the candida species. Figure 4 displays a really comparable antifungal movement by all the test compounds, there are numerous components increment the movement are bond length among the metal, conductivity, dipole minute, and dissolvability [22-25].



**Figure 3:** The inhibition zone of  $[M(L)(L-dopa)_2]$  against *Staphylococuse* and *Klebsiell*



**Figure 4:** The inhibition zone of  $[M(L)(L-dopa)_2]$  against *candida albicans*

## Conclusion

In this study, a Schiff base ligand (Curcumin with 3-aminophenol) was synthesized. The stable mixed ligand complexes,  $[L-M-(L-dopa)]$  system was formed with transition metal ions such as Mn(II), Cu(II), Ni(II), Zn(II), Cd(II), and chlorid, and then they were synthesized. The activity of the synthesized mixed Schiff curcumin-L-dopa

complexes matches the effectiveness of some anti-microbial strain.

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## Authors' contributions



All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work.

### Conflict of Interest

The author declared that they have no conflict of interest.

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### Supporting Information

Our work includes the synthesis of new compounds, which is consistent with the goals of the journal and adds new scientific parameters to the researchers (PDF).

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